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A. A. BRENEMAN, 97 Water St., New York.

PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

REGULAR MEETING, November 4th, 1887.

Mr. James H. Stebbins in the Chair.

The minutes of the preceding meeting were read and approved.

Dr. Cyrus Edson and Mr. H. F. Starr were unanimously elected members.

A Paper was read by Dr. W. H. Kent on "A modified form of Sohxlet's Apparatus for the Determination of Fat in Milk," and one by Prof. W. P. Mason "On the United States Gallon."

The Committee on Nominations presented a ticket for the election of officers for 1888.

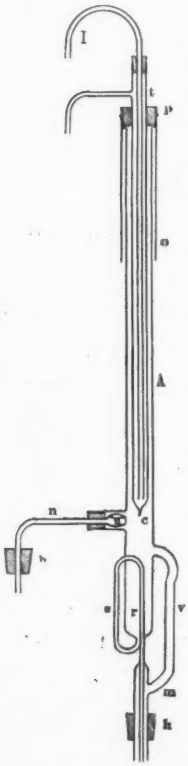
The meeting was then adjourned.

T. D. O'CONNOR,
Recording Secretary.

ON A MODIFIED FORM OF SOXHLET'S EXTRACTOR.

By Walter H. Kent, Ph.D.

In the examination of milk in the Health Department of Brooklyn, we have had occasion to try some of the various methods for the determination of fat in milk. In 1885, while the American Society of Public Analysts was endeavoring to fix upon some method, Dr. Otto Grothe and myself made comparative determinations by some of the methods, the results of which have already been published.*



While engaged in this work the form of extractor here described, was devised and first constructed by Dr. Grothe. With some minor improvements, which I have been able to introduce, this extractor has since been used and with much satisfaction.

It will be seen to be a combination of the Soxhlet extractor with an external condenser, as described by Dr. R. Wollny.†

When in use the extractor is attached by a clamp at *A* to a firm support. The support I use consists of an extension of the gas pipe from the table to the ceiling of the room, where it is secured by passing through the wooden ceiling. The condenser, the lower point of which is seen at *c*, rests by the cork *p* on the upper end of the outer tube *A*, the cork being concave below so as to keep the condenser in the center. The condensing tube *t* is firmly joined by the cork *p* to the outer tube *o*, by which the point *c* is also held in the center, if necessary, by the aid of a little wedge of wood between the tube *A* and the lower end of the tube *o*.

* Annual Report of the Department of Health of Brooklyn, N. Y,. 1885, p. 113; London *Analyst*, 1885, p. 47.

† Fresenius Zeitschrift, 1885, p. 106.

In introducing the dried milk into the receptacle *r*,* the clamp at *A* is loosened and the lower part removed, while the condenser is supported at *t* by a string to the support above. As I have it arranged a projecting ring from the gas pipe also serves to support the condenser.

As a means of introducing and withdrawing the tube containing the substance to be extracted, a white thread, previously treated with ether, may be connected with the tube; during the extraction this thread extends over the tube *A* and out under the tube *o*. The tube *n*, for finally withdrawing the ether, is sufficiently large to allow the ether to flow through without obstructing the tube; any ether vapor from the receiving flask at *b* can thereby pass upward into the condenser. The flask serves by the cork *b*, to close the tube *n* during the withdrawal of the ether by the spoon *x*, while the tube *n* also serves the double purpose of supporting the flask by the cork *b*. If the flask at open and supported by other means, the ether vapor from the tube *b* is left *r* passes out at *n*, without being forced up into the condenser, and is thereby lost.

The flasks used hold about 150 c.c. They have a short, wide neck and all fit the same cork. The corks *b* and *h*, being of the same size, the flasks may be used interchangeably. The weighed flask at *b*, serves to close the tube *n* during one extraction, and, after finally receiving the ether, is ready to take the place of the fat receiving flask in the next extraction. A third flask may take the place *b*, and thus another extraction is made while the fat of the first extraction is being dried. In constructing the extractor, the syphon tube *s* should be small, so as to fill readily with ether as it flows over the upper bend of this tube. The vapor tube *r* should be sufficiently large to permit the upward passage of the vapor without being obstructed by the ether that condenses in this tube. A pocket in the tube at the bend *m* should also be avoided.

Of the various materials used as media on which to dry the

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* In the drawing the syphon tube *s* extends downward in front of the receptacle *r* of the main tube *A*.

milk, I much prefer asbestos, used as described by Dr. S. M. Babcock.*

The size of the tube for the asbestos may be regulated by the size of the receptacle *r* of the extractor. The perforation in the bottom of the tube should be large enough to allow a rapid discharge of the ether. A tuft of cotton, which has been purified by extraction with ether, covers the hole in the bottom of the tube, over which about two inches of the tube is gently packed with ignited asbestos. Another tuft of cotton, which may be removed for the introduction of the milk, serves to cover the mouth of the tube and to retain the asbestos. The milk may be weighed in the asbestos tube thus prepared, the tube being held upright by the support, with which balances are often provided. During the weighing and drying of the milk it is an advantage to keep the tube upright, as by so doing the milk is more uniformly distributed over the asbestos.

For a holder of the tube while drying, a small beaker may be provided with a doubly perforated, tightly fitting cork. Through one perforation the asbestos tube passes to near the bottom of the beaker, while a tube through the other perforation serves to connect with the pump. Instead of beakers I use cupping glasses; these are stronger and therefor permit the cork to be more tightly fitted by pressure.

I prefer the asbestos method to that using paper, as described by Adams,† and afterwards adopted by the English Society of Public Analysts for the following reasons:

1st. The asbestos is more readily prepared than paper. The tubes are easily made by a chemist and may be used any number of times. The small amount of cotton employed can be purified in the extractor. The cotton and the ignited asbestos are kept in bottles ready for use. This, I think, is less trouble than to purify by extraction the relatively large volume of paper necessary in the Adams method. With reference to purifying the paper P. Vieth

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* Second Annual Report of the N. Y. Agricultural Experiment Station, p. 168; Report the Department of Health, of Brooklyn, 1885, p. 112; U. S. Department of Agriculture, Division of Chemistry, *Bulletin* No. 16, p. 76.

† London *Analyst*, 1885, p. 46.

says:* "It is indispensable that the paper used in the process should be most thoroughly exhausted from matter soluble in ether."

2d. Babcock's method I consider more accurate. In the use of asbestos I have not known of the question of accuracy being raised. This cannot be said with reference to the paper method. Unlike asbestos, paper is an artificial substance; while it consists principally of cellulose, it may contain various other organic substances. At the meeting of the Association of Official Agricultural Chemists, in August last, Dr. W. McMurtrie said that he had found in the paper used by him "a waxy, resinous substance, with difficulty soluble in ether."† In this case it is precisely those substances which are soluble with difficulty that are to be feared. Were they easily soluble they would be easily removed. In making the Adams method an official method the above association says: "It gives uniformly about 0.2 per cent. more fat in normal milk than the ordinary gravimetric methods."‡ Dr. Vieth made comparative determinations of fat in sixteen samples, using the plaster of paris and paper methods. He continues as follows (*loc. cit.*): "On comparing the results of the two methods it was found that in all and every case a higher percentage of fat was obtained by using the paper method, the plus ranging from .07 to .46, and averaging .271."

From this it would appear that there is a considerable uncertainty in the process, and that a maximum error of nearly one-half per cent. may result. From the complex and uncertain composition of paper it could not be expected that this discrepancy would be uniform.

3d. An advantage in the use of asbestos arises from the secure condition of the milk, after it is once introduced into the tube and during the process of drying. It may be handled in any manner, and nothing short of rupture of the tube will lead to loss; and

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* London *Analyst*, 1887, p. 62.

† U. S. Department of Agriculture, Division of Chemistry, *Bulletin* No. 16, p. 30.

‡ U. S. Department of Agriculture, Division of Chemistry, *Bulletin* No. 16, p. 74.

further, as Dr. Babcock says, after once heating, the milk is sterilized and the process of drying and extracting may be completed at any subsequent time.

4th. The total solids may be determined on the ignited asbestos without the further trouble of previously drying to a constant weight.

Some recent experiments on the necessary amount of drying and extracting will be given in another paper.

THE UNITED STATES GALLON.

"The gallon is a vessel containing 58372.2 grains (8.3389 pounds avoirdupois) of the standard pound of distilled water, at the temperature of maximum density of water, the vessel being weighed in air in which the barometer is 30. inches at 62° F." —(See report on "Weights and Measures," by Secretary of the Treasury, Senate Doc., 1857.)

This definition being somewhat obscure, occasion was taken to write the Treasury Department, from which letter the following is quoted:

"Does this wording mean that the '*gallon*' is *a volume* equal to that occupied by the above quantity of water under the conditions named; or does it intend a '*gallon*' to be understood as meaning the quantity of liquid capable of being held by such standard *vessel* as above referred to, even after said vessel has been expanded by elevation of temperature? I take it that the latter view, although certainly stated, can hardly be intended."

The reply to this came from the office of the "Coast Survey," and read as follows:

"The standard gallon is a measure of capacity or volume. Its capacity was derived from standard weights, and in verifying and standardizing capacity measures it is the practice to determine the volumes by weighings. It is, therefore, to be understood that the capacity of a gallon is measured by the weight of a volume of distilled water at maximum density, whose weight in air at 62° F. Bar. 30. inches, equals 58372.2 grains.

"It follows, therefore, that a gallon is the measure of a constant volume, and that due allowance must be made for changes of temperature of a vessel used as a gallon measure."

A letter recently coming from the Treasury Department reads: "The value adopted by this office for the weight of 231 cubic inches of distilled water at its maximum density is 8.3389 lbs.: or 8.3316 lbs. at 15° C. This reduced to 60° F. gives a value of 8.3312 lbs."

Desiring to obtain a value in pounds for a U. S. Gallon of distilled water at 60° F., said value to be carried beyond four decimal places, a very surprising degree of confusion was discovered among the authorities.

For instance:

U. S. Pharmacopœia, 1870	58328.8862 grains or	8.332698	lbs.
" " 1880	58329.6 " "	8.3328	"
Miller's Chemistry	58317.3 " "	8.3310	"
Am. Chemist, vol. 1, page 318	58319.8 " "	8.3314	"
U. S. Treasury Depart	——— " "	8.3312	"
" Dispensatory (last ed.)	58328.886 " "	8.332698	"
Oldberg's "Weights and Measures," page 167	58335.218 " "	8.333602571	"

From Barnard's "Metric System" we have:

1. cubic inch pure water at 62° F., and under a pressure of 30. inches of mercury weighs *in vacuo* 252.75965 grains.

Using Kopp's tables (Watt's Dict., vol. 3, p. 58, and interpolating by formula number one), we have:

1 cu. in. water at 22° F. becomes:

At 60° F	1.000769074769 cu. in.
" 62° "	1.000942703386 " "

Hence:

1 cu. in. at 62° F. becomes .9998265349101 cu. in. at 60° F.

Hence:

1 cu. in. of water at 60° F. weighs *in vacuo* 252.8035026 grains.

And 1. gallon (232 cu. in.) *in vacuo* { 58397.6091006 grains or 8.3425155858 lbs.

Referring again to Barnard's "Metric System," we find:

1 cu. in. water at 62° in air at 62° weighs.....252.48843 grains.
1 " " dry air at 62°..... " ..... 0.30908093 "
1 " " " " " 60°..... " ..... 0.3102675 "

Hence:

1 cu. in. water at 62° *in air at* 60° " .....252.48724343 "

Now 1 cu. in. of water at 62° becomes, as we have seen:

0.9998265349101 cu. in. at 60°; and the weight of this volume of water at 60° is equal to that of the entire cubic inch at 62°, plus the weight of a volume of air at 60°, equal to the difference between unity and said volume of water.

Hence: .9998265349101 cu. in. water at 60° and in air at 60° weighs 252.48729724 grains.

Hence: 1 cu. in. water at 60° weighed in air at 60° and under 30 inches mercury weighs 252.53110257 grains.

From these results we obtain for the weight of one U. S. Gallon (231 cu. in.) of pure water at 60° F., and weighed in dry air at 60° F. under a pressure of 30 inches of mercury:

58334.68469367 grains or 8.33352638481 lbs.

WILLIAM P. MASON.

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TROY, N. Y.

ABSTRACTS.

GENERAL AND INORGANIC CHEMISTRY.

Dehydration of Metallic Hydroxides by Heat. T. CARNELLEY AND DR. J. WALKER.

A very long and full article, containing many tables, and largely illustrated by diagrams.

Among the "general conclusions" of the authors may be noted:

The action of heat on the hydrate of PbO_2 will possibly be of interest in the manufacture of red lead; hydrate dried in air=3 PbO_2H_2O, complete dehydration at about 230°; PbO_2 is stable up to about 280°; loss of oxygen with formation of Pb_2O_3, at 280°–290°; Pb_2O_3 stable 290°–360°, loss of oxygen with formation of Pb_3O_4, 360°–415°; Pb_3O_4 stable 415°–530°, loss of oxygen with formation of PbO at 530°–580°; PbO stable from 580° to above 815°; PbO fused somewhere between 585° and 630°.

The retention of water by many oxides at comparatively high temperatures, at and above a red heat, is noteworthy. This is especially interesting in the case of the hydrate of cerium dioxide, $Ce(OH)_4$, which is stable to as high a temperature as 600°.

The retention of water at such high temperatures illustrates the great importance in quantitative analysis of the thorough ignition of hydrates in order to drive out the last traces of water before weighing.

The minimum temperature of complete dehydration is a periodic function of the atomic weight, as follows:

(*a.*) For normal oxides of *odd* members of the same group, the minimum temperature of complete dehydration diminishes as the atomic weight of the positive element increases. (No exception.)

(*b.*) For *even* members of the same group it increases, as the atomic weight of the positive element increases. (No well marked exceptions.)

(*c.*) For normal oxides of elements belonging to the same *period*, the minimum temperature of complete dehydration diminishes from the beginning to the middle, and then increases to the end of the period. The only apparent exceptions are MgO,Bi_2O_5 and I_2O_7(?).

Changes of color on dehydration, which are *permanent on cooling*, usually indicate the formation of a definite hydrate, or that the dehydration is complete, or that some other definite chemical change has occurred. (*Jour. Chem. Soc.*, ——*59.*) W. P. M.

Charcoal prepared by Superheated Steam for the Manufacture of Gunpowder. L. RAMDOHR.

By none of the ordinary methods of making charcoal can an entirely uniform product be obtained, but by the use of superheated steam a definite and regulated heat may be applied. The chief advantages are: (1) The carbonization can be followed through each stage, whereby charcoal from the clearest red-brown to the deepest black may be obtained. (2) Since the steam penetrates the carbonizing wood uniformly, the resulting charcoal is absolutely uniform. (3) The tarry products of distillation, which by all other methods of carbonizing, undergo a partial decomposition in the retort to the injury of the charcoal, are immediately removed from the retort without decomposition. (*Chem. u. Tech. Ztng.*, **1887,** 588.) D. W.

Comparative Antiseptic Values of Chlorides, Nitrates and Sulphates. C. T. KINGZETT.

Experiments made to determine for what period certain quantities of 5 per cent. solutions of different chlorides, nitrates and sulphates would protect flour paste from mold, and by similar trials beef extract from putrefaction.

Compounds of the alkalies and alkaline earths do not prevent but seem to hasten the appearance of the mold in flour paste, metallic compounds, except zinc, delay it considerably, and the chlorides of lead, copper and mercury, besides nitrate of copper and sulphate of mercury, show the highest efficiency.

With beef extract, copper and mercury compounds are the most effective for the prevention of putrefaction, the chlorides being

most active. Zinc salts appeared to be more powerful in preventing the putrefaction of the beef extract than in preventing the growth of mold. (*J. Soc. Ch. Ind.*, **6**, 702.) C. F. McK.

ORGANIC CHEMISTRY.

Synthetical Experiments in the Sugar Group. E. FISCHER and J. TAFEL.

As has been previously shown by the authors, acrolein bromide is converted by baryta into compounds, which, in their behavior, very much resemble the sugars. They are isolated by means of phenylhydrazine, with which they yield α and β-phenylacrosazones, which melt at 205° and 148° respectively.

By the reduction of α- phenylacrosazone, a base, α- acrosamine, which is isomeric with isoglucosamine, is produced, and this, when treated with nitrous acid, gives a non-nitrogenous, syrupy body, possessing all the properties of the sugars, differing, however, from the natural sugars in being optically inactive.

Phenylacrosazone, $C_{18}H_{22}N_4O_4$ is prepared by adding 50 grms. of acrolein bromide, freshly distilled in vacuo, drop by drop, to a solution of 75 grms. barium hydroxide in 1.25 liter of water, which is carefully cooled in ice-water, and violently shaken. The solution obtained by eight operations is slightly acidified with sulphuric acid, and the barium precipitated with a concentrated solution of sodium sulphate. The solution is filtered, exactly neutralized with sodium hydroxide, and evaporated in vacuo on a water bath. After cooling, a solution of 50 grms. phenylhydrazine hydrochloride, and 50 grms. crystallized sodium acetate in 100 cc. water is added. At the end of 12 hours a reddish brown resin separates. The filtrate receives a further addition of 150 grms. phenylhydrazine hydrochloride, and 150 grms. sodium acetate, and is then heated on the water bath. After about four hours the dark semicrystalline precipitate which

separates is washed with water, and dried on porous plates. By treating with ether the greater portion of the β- phenylacrosazone and resin are dissolved, while the α- acrosazone remains, in the dark colored residue. This residue is several times treated with alcohol, and then with boiling water. The product, which now has a yellow color, is almost chemically pure. 400 gms. acrolein bromide yield on an average about 18 grms.

α- *Acrosamine*, $C_6H_{13}NO_5$, is obtained by reducing the acrosazone with zinc dust and acetic acid. The base shows all the reactions of glucosamines. It reduces Fehling's solution, and turns brown when heated with alkalies, giving off ammonia. Nitrous acid converts it into glucose.

β- *Phenylacrosazone* is obtained from the ethereal solution resulting from the purification of the α- compound, although in the pure state it is insoluble in ether. The ether is evaporated, the residue dissolved in alcohol, and precipitated with water. The filtered product is exhausted with cold benzene, when a yellow crystalline compound remains. (*Ber. d. Chem. Ges.*, **20**, *2566.*)

W. R.

Oxidations by Means of Hydrogen Peroxide. C. WURSTER.

Hydroxylamine sulphate is quantitatively oxidized to sulphuric acid and nitric acid by hydrogen peroxide, when heated to 40°:

$$(H_2NOH)_2.H_2SO_4+6H_2O_2=H_2SO_4+2HNO_3+12H_2O$$

Under the same conditions hydroxylamine chloride is oxidized to hydrochloric acid and nitric acid.

If to an aqueous solution of phenol a salt of hydroxylamine and hydrogen peroxide are added, a yellowish coloration slowly takes place in the cold. At a temperature of 40° the characteristic coloration of nitrosophenol is rapidly produced. The nitrosophenol may be obtained in brown crystals by shaking out the solution with ether, and evaporating the latter.

Phenylhydrazine is at first dissolved by hydrogen peroxide. Subsequently the solution becomes turbid and yellow, and a strong evolution of gas takes place. By distillation with steam a yellow oil passes over, which on fractional distillation was found to consist of benzene, and diazobenzeneimide. (*Ber. d. chem. Ges.*, **20**. *2631.*)

W. R.

Orthothioxen and Orthothiophendicarboxylic Acid. W. GRUENEWALD.

Orthothioxen $C_4H_2S(CH_3)_2$ is prepared by distilling an intimate mixture of 10 grms. β- methyllevulinic acid with 17 grms. phosphorus trisulphide, in a capacious retort. A gentle heat is sufficient to induce the reaction, which then completes itself. The resulting oily product is purified by fractional distillation, when about one-third, consisting essentially of thioxen, passes over between 120° and 150°. This fraction was boiled with sodium hydroxide for three hours, and then fractioned over sodium.

Pure orthothioxen forms a colorless, strongly refractive oil, having an odor like petroleum; boiling at 136°-137°, and having a specific gravity of 0.9938 at 21°.

The thioxen was oxidized with a 1% solution of potassium permanganate, in sufficient quantity to oxidize both methyl groups, but only a monocarboxylic acid was obtained. By oxidizing this acid alone a mixture of mono- and dicarboxylic acid resulted. Orthothioxencarboxylic acid, $C_4SH_2(COOH)_2$ crystallizes in long needles. It does not melt at 260°, but is decomposed at a higher temperature. When heated with resorcinol for some time to 200°, a melt is obtained, which in concentrated alkaline solution has a dark red color, changing to yellow when diluted and showing a fine, yellowish-green fluorescence. The silver salt forms a white, flocculent precipitate, insoluble in water. The lead salt, separated from a solution of the ammonium salt, also forms a white, flocculent precipitate. The barium salt crystallizes in colorless crystals containing no water or crystallization. The dimethyl ether, obtained from the silver salt by heating with methyl iodide, crystallizes from alcohol in colorless plates, melting at 59.5°. (*Ber. d. Chem. Ges.*, **20**, *2585.*) W. R.

Derivatives of Di-β-naphthylamine. CHR. RIS.

The dinaphthylamine was purified by distillation, and recrystallization from benzine. Boiling point about 471°.

Methyl-and ethyl-β-dinaphthylamine $(C_{10}H_7)_2NCH_3$ and $(C_{10}H_7)_2NC_2H_5$ were prepared. They both crystallize in colorless needles and distil without decomposition. The melting point of the methyl compound is 139–140°, that of the ethyl compound

130°. The basic properties are faint. The salts are decomposed by water.

Methyldi-β-naphthylcarbamate $(C_{10}H_7)_2NCO_2CH_3$, prepared by heating the secondary amine with methyl chloroformate above 130°. Crystallizes from benzene in white, warty groups, containing ½ mol. benzene, from alcohol in fine needles. Melts at 113–114°. Distils almost without decomposition.

Tetrabromdi-β-naphthylamine, $C_{20}H_{11}Br_4N$, prepared by the action of an excess of cold bromine on di-β-naphthylamine. Long felted needles, sparingly soluble in most solvents, and melting at 246°.

Octobromdi-β-naphthylamine, $C_{20}H_7Br_8N$. Prepared by the action of bromine in presence of aluminium bromide. Fine white needles, sparingly soluble and melting above 360°.

Nitrosodi-β-naphthylamine $(C_{10}H_7)_2NNO$, obtained by the action of nitrous acid on di-β-naphthylamene. White groups of needles melting at 140°.

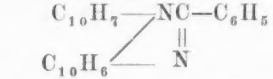
The corresponding hydrazine could not be obtained.

Dinitrodi-β-naphthylamine, $C_{20}H_{13}(NO_2)_2N$. Yellowish red needles, slightly soluble, melting at 224–225°.

Tetranitrodi-β-naphthylamine, $C_{20}H_{11}(NO_2)_4N$. Yellow crystalline grains. Soluble in boiling nitrobenzene. Melting point 285–286°.

Mononitrobenzoyldi-β-naphthylamine, $C_{27}H_{18}(NO_2)NO$, obtained by nitrating benzoyldi-β-naphthylamine. Crystallizes from benzene in yellow transparent prisms with 1 mol. benzene, from alcohol in yellowish crystals, melting at 168°.

Benzenylnaphthylnaphthylenediamine,



is obtained by the action of reducing agents on the previous compound. The anhydrobase crystallizes from benzene in fine needles or prisms. Melts at 163°. May be sublimed, and distils with but little decomposition. (*Ber. d. chem. Ges.*, **20**, *2618.*) W. R.

Action of Acid Amides on Bromacetophenones. M. LEROY.

When bromacetophenone is heated with two parts acetamide for about one hour to 120–130°, a base is obtained which crystallizes in long needles from its solution in alcohol and ether, melts at 45°, distills without decomposition at 241–242, and has the composition $C_{10}H_9NO$. The hydrochloride crystallizes in small needles. The platinochloride forms fine orange yellow needles, which melt at 130–140°. The sulphate crystallizes in white plates, having a pearly luster, and is decomposed by contact with cold water.

The formamide base is prepared by heating bromacetophenone with formamide to 120–130°. It has the composition C_9H_7NO, forms a thick, colorless oil, has a peculiar odor, and crystallizes in stellar crystals when cooled by a freezing mixture. These crystals melt at 6°, and distil at 220–222°. The hydrochloride forms a white crystalline mass, melting at 80°. The platinochloride crystallizes in fine yellow needles.

The benzamide base, $C_{15}H_{11}NO$, is obtained by melting together bromacetophenone and benzamide at 140–150°. It crystallizes from alcohol in large colorless plates, melts at 102–103°, boils at 338–340°, and is readily soluble in warm alcohol, ether, benzene, etc. The hydrochloride crystallizes in white needles. (*Ber. d. Chem. Ges.*, **20**, *2576.*) W. R.

Isonitroso Compounds. E. BECKMANN.

In a previous paper the author has expressed the opinion that the molecular changes produced by the action of sulphuric acid on ketoximes, might also be effected by other acids. This is now shown to be true, especially for hydrochloric acid, acetyl chloride, acetic anhydride and acetic acid. (*Ber. d. Chem. Ges.*, **20**, *2580.*) W. R.

The Three Isomeric Pyrocresols. W. BOTT.

The crude pyrocresols are obtained from certain kinds of acid oils or phenols by extraction with caustic soda and fractionation of the free phenols obtained by acidifying the alkaline extract. The three pure isomerides, when separated, take up one atom of oxygen to form the oxides. Halogen and nitro- and sulpho-com-

pounds were also obtained. Judging from experiments not yet concluded it is probable that the empirical formula $C_{15}H_{4\,0}$ is correct, and that the three isomerides are anhydrides like dipheynl ether, formed of two chains united by oxygen $C_xH_xC—O—CC_yH_y$. (*Jour. Soc. Chem. Ind.*, **6**, *646.*) C. F. McK.

Condensation Product of Cinnamic Acid and Gallic Acid. E. Jacobsen and P. Julius.

Styrogallol, $C_{16}H_{10}O_5$, is prepared by heating 10 parts cinnamic acid and 17 parts gallic acid with 150 parts sulphuric acid for 2–3 hours to 45–55°. It is soluble in all ordinary solvents, excepting boiling alcohol, glacial acetic acid and aniline; in the last mentioned it is sparingly soluble. It crystallizes in pale yellow, microscopic needles, does not melt at 350°, and sublimes without decomposition in fine, large, brilliant yellow needles. It dissolves in alkalies with a green color, which on heating passes through blue and violet to red. In concentrated sulphuric acid it dissolves with a fine yellowish red color.

Styrogallol may also be prepared by using the corresponding weight of tannic acid in place of the gallic acid.

A triacetyl derivative has also been obtained by treating the styrogallol with acetic anhydride. (*Ber. d. Chem. Ges.*, **20**, *2588.*) W. R.

Phenylated Piperidine and Pyridine Bases. O. Bally.

γ- Phenylpiperidine, (γ) C_6H_5, $C_5H_{10}N$ can be prepared quantitatively from γ- phenylpyridine. It boils at 255–257°, and when freshly distilled melts at 57.5–58°. It is a strong base, barely soluble in water, and absorbs carbon dioxide from the air. Its salts are readily soluble.

Symmetrical Phenyllutidine, C_5NH_2, αCH_3, γC_6H_5, αCH_3. By saponifying the phenyllutidine dicarboxylic ether obtained from benzaldehyde, acetacetic ether and ammonium hydroxide with alcoholic potash and heating the product with lime a greenish yellow oil passes over which solidifies after a time, and dissolves in ether with a greenish blue fluorescence. The base was purified by solution in hot hydrochloric acid, boiling the crystallized hydro-

chloride which separated on cooling in aqueous solution with bone black, and extracting the filtrate, after making it alkaline, with ether. From this solution, which no longer fluoresces, the base crystallizes in prisms, melting at 54.5–55°. It boils at 287° under 731 mm. pressure.

γ- Phenyllupetidine, $C_6H_5C_5H_8(CH_3)_2N$ is a colorless oil, boiling at 274° under 731 mm. pressure, which does not solidify at —16°. (*Ber. d. Chem. Ges.*, **20**, *2590.*) W. R.

Chemical and Therapeutical Study of Salol. DR. LOMBARD.

Salol is a direct combination of salicylic acid and phenol with elimination of water.

$$C_6H_5OH+C_6H_4OH,COOH=C_6H_4OH,COOC_6H_5+H_2O.$$

It is prepared by the reaction of phosphorus perchloride, carbon oxychloride, or HCl in the nascent state on a mixture of sodium salicylate and phenate. It has the appearance of a white, unctuous powder; insoluble in water, glycerol and liquid vaseline, soluble in ether, chloroform, benzine, essence of turpentine, and the volatile or fixed oils. It is very soluble in alcohol, melts at 42°—42.5°, and remains in a superfused state from which it recrystallizes on agitation.

This body is isomeric with the salicyphenol of Michael prepared by the action of stannous chloride on a mixture of phenic and salicylic acid, and represented by Michael by the formula

$$CO.\ C_6H_6.\ OH_2.$$

Salol is not poisonous and is decomposed in the duodenum into phenic and salicylic acids; its effects have been very salutary in the treatment of rheumatism, and in internal application. (*Arch. de Pharm.*, **2**, *388.*) M. L.

ANALYTICAL CHEMISTRY.

Analysis of Different Varieties of Coals. G. KLIEBHAN.

Charcoal.—Charcoal consists of carbon, potash and lime salts and variable quantities of water. Determine water in usual manner, drying at 100° till weight is constant.

Determine ash by ordinary method. Treat ash with water; solution contains potassium carbonate, sulphate, silicate and chloride. Concentrate solution and divide in three or four parts. In one, determine carbonic anhydride and calculate to potassium carbonate; in another sulphuric acid and calculate to potassium sulphate; in another chlorine, for potassium chloride. Silica is determined by extracting a weighed portion of the ash several times with nitric acid diluted with two volumes water. Residue is silica. The nitric acid solution may contain calcium phosphate and oxide of iron. Evaporate to dryness, dissolve in water, precipitate iron by ammonium sulphide; evaporate filtrate to dryness, ignite and weigh; calcium phosphate.

Bone Charcoal.—Carbon containing hydrogen, nitrogen, calcium phosphate and carbonate, small quantities of sodium chloride, carbonate and phosphate; magnesium phosphate, oxide of iron, silica and alumina. Determine moisture at 120° in oil bath.

Dried coal is ignited, ash dissolved in dilute hydrochloric acid, and any residue counted as impurity. Calcium and barium phosphates are precipitated by ammonia, dried and weighed. In filtrate from phosphates, precipitate lime by sodium carbonate.

Extract a weighed quantity of the bone charcoal with hot water, evaporate to dryness and weigh total salts, then analyze by usual methods. Determine nitrogen by combustion with copper oxide, as usual.

Peat.—[TORFKOHLE.] The carbon is combined with some hydrogen and nitrogen. The ash contains silica, alumina, oxide of iron, lime and magnesia. The analysis of these is as above described. The residues insoluble in water are treated with hydrochloric acid, whereby silica and alumina remain behind.

Pit Coals.—The varieties of coal here included consist of carbon, hydrogen, oxygen, in different proportions ; sometimes also, nitrogen. Many contain alumina and ferrous carbonate ; most of them contain pyrites which in any quantity is especially injurious to the coal as a fuel. Determine ash by ignition in the air ; pyrites by treating the finely powdered coal with hot aqua regia ; the sulphur is converted into sulphuric acid, the iron into oxide. Determine either the iron or sulphuric acid, or both, and calculate quantity of pyrites present in the coal. In the same manner ferrous sulphide may be determined in coke.

Carbonaceous Shale.—[SCHIEFERKOHLE] Is an easily friable coal used in large quantity as a decolorizer. The analysis is effected as already described. After the determination of hygroscopic moisture by drying in oil bath the dried coal is boiled with water to dissolve calcium sulphate, the quantity being determined by weighing the again dried and lime-free coal. The coal thus exhausted by water is treated with rather strong, cold hydrochloric acid, whereby calcium carbonate goes into solution, while aluminium silicate remains undissolved. The chief difference between shale coal and bone charcoal is in the presence in the latter of phosphoric acid. Also the ash of contains silica, alumina and oxide of iron which never occur in bone charcoal. The last circumstance furnishes the means of distinguishing the adulteration of bone charcoal with shale coal without the aid of the microscope. (*Chem. u. Tech. Ztng.*, **1887**, 551.) D. W.

Detection of Minute Quantities of Carbon, Dioxide and other Gases. O. ROESSLER.

A small test tube is drawn out to a capillary at the lower end, and bent upward. At a distance of about 1 c.m. from the bend it is cut off. Another thin glass cylinder, fitting into the first, is drawn out to a capillary funnel, the upper portion of which fits into the neck of the first, while the point reaches to 1.5–2 c.m. from the bottom of the bent tube.

The substance under examination is placed into the dry bent tube. Then the capillary funnel, filled with as much baryta water as will be held by the surface tension, is inserted. A small

drop of the liquid will then hang at the lower end without any tendency to drop off. If then the lower portion of the apparatus is immersed in hydrochloric acid, the latter will enter through the capillary and liberate the carbon dioxide, which will cause a turbidity in the baryta water.

To detect sulphurous acid, nitric acid, hydrogen sulphide, nitrous acid, ammonia, etc., the capillary funnel is filled with iodide of starch, ferrous sulphate, lead acetate, potassium iodide, cupric sulphate, etc., respectively.

The author found that by means of this apparatus he could detect the presence of 0.02 mgrm. carbon dioxide. (*Ber. d. chem. Ges.*, **20**, *2639.*) W. R.

Separation of Stannic Oxide from Tungstic Acid. E. DONATH AND F. MULLNER.

A method applicable to bronzes and other tin alloys containing wolfram. It depends on the reduction of stannic oxide by ignition with finely divided zinc, to a sponge of metallic tin, readily soluble in hot, dilute HCl, while tungstic acid, by this treatment, is reduced only to blue tungstic oxide, which by oxidation is easily converted into tungstic acid, insoluble in HCl. The finely divided zinc, either as dust, powder or filings, must be proven pure before use. It may be most readily obtained in suitable condition by filing a rod of pure zinc, removing particles of iron by a magnet, until no reaction for iron can be obtained. The weighed and ignited mixture of stannic oxide and tungstic acid, obtained by the usual treatment of the alloy with HNO_3 is triturated with twice its volume of zinc filings in an agate mortar, transferred to a porcelain crucible, covered and strongly ignited for fifteen minutes. After cooling, the spongy mass is transferred to a beaker, treated with diluted HCl (1 to 2) and boiled till no more H is given off and the tin is completely dissolved. After cooling, powdered $KClO_3$ is carefully added until the blue tungstic oxide is oxidized to yellow tungstic acid, and the solution is absolutely free from blue color; it is diluted with $1\frac{1}{2}$ volume of water and allowed to stand 24 hours. The separated tungstic acid is then filtered off, washed with water acidified with HNO_3 and finally with a hot dilute solution of

ammonium nitrate to avoid cloudy filtrate—dried, ignited and weighed as tungstic acid, and the tin oxide calculated by difference.

For direct estimation of the tin, it is only necessary to precipitate the filtrate from the tungstic acid by H_2S, and to convert the sulphide into stannic oxide by prolonged ignition with access of air, or with the aid of a little ammonium nitrate. This direct estimation of tin cannot be made with accuracy in this way if the zinc used in the reduction contained cadmium. Results of five experiments are given to prove the accuracy of method. (*Zeit. Berg-u-Huttenwesen*, **1887**, *614.*) D. W.

Detection of Aniline Colors in Wines, Fruit Juices, etc. C. O. CURTMAN.

Hoffman's isonitril test for chloroform has been successfully used by the author to detect aniline colors in wines, etc., even when the coloring matter was present in only minute quantities. To the wine, or to the prepared alcoholic solution of the coloring matter, add solution of potassium or sodium hydrate and a few drops of chloroform, heat gradually for about a minute and finally boil. The isonitril odor will be readily detected. In some cases the reaction is intensified by adding an excess of sulphuric acid to the boiled alkaline solution. (*Fres. Zeit.*, **26**, *555.*) J. F. G.

Detection of "Fahlberg's Saccharin"—Ortho-Sulphaminbenzoic Acid. C. SCHMITT.

The product obtained by the Fahlberg process in the manufacture of "saccharin" is ortho-sulphaminbenzoic acid, having the composition $C_6H_4.CO.SO_2.NH$. The extreme sweetness of this compound and its antiseptic properties suggest its use in foods.

To identify saccharin in wine, extract the same with ether. Evaporate the ether solution, dissolve the residue in hot water, add an excess of lead acetate, and remove excess of lead with dilute sulphuric acid after filtration. Heat the filtrate and neutralize with barium carbonate and filter. In the evaporated filtrate the saccharin may be recognized by its extreme sweetness and the application of chemical tests.

1. Saccharin heated with concentrated hydrochloric acid produces ammonium chloride; and 2, when fused with an alkali and oxidizing agent it yields sulphate. These reactions are only confirmatory. For fusion a mixture of sodium carbonate and potassium nitrate (6:1) may be used. The most conclusive test is the formation of salicylic acid, as suggested by the author. Saccharin, when heated with an alkali, yields salicylic acid as one of the products. By the following process as little as .005 per cent. saccharin can be detected in wine:

Strongly acidulate 100 c.c. of wine and extract with three portions, 50 c.c. each, of a mixture of equal parts of ether and petroleum ether. Mix the separated ether extracts and add an excess of solution of sodium hydrate, evaporate to dryness and heat the residue for about ½ hr. at 250° C. Dissolve the fused mass in water, acidulate with sulphuric acid, and extract the solution with 50 c.c. of ether. Exaporate the ether, take up with hot water and test with dilute ferric chloride solution, which will give the characteristic purple with the salicylic acid formed. Tannin and the astringent matter of wine may yield traces of salicylic acid on fusion with an alkali hydrate, but the author overcomes this difficulty by the use of the mixed ethers in which the tannin, etc., is insoluble. The presence or absence of salicylic acid in the wine must be previously determined. (*Rep. d. a. Chem.*, **7**, *437-441.*)

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